

## A Tribenzylidenemethane–Tantalum Compound: Some Experiences with ‘Inversion Twinning’

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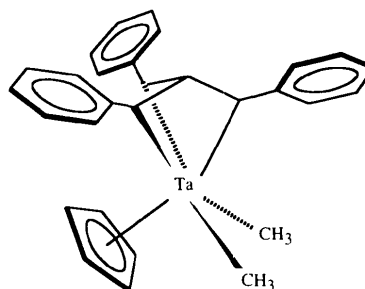
### Abstract

The six-electron-donating ligand tribenzylidenemethandiide has been used to form a tantalum (group 5) mimic, ( $\eta^5$ -cyclopentadienyl)( $\eta^4$ -tribenzylidenemethandiide)dimethyltantalum, of a group 4 bent metallocene. The material crystallizes with two molecules in the asymmetric unit with quite different packing arrangements, although the overall structures of the two are similar. The Cp and methyl ligands are disordered about a three-fold axis. Crystal data:  $[\text{Ta}\{(\text{C}_7\text{H}_6)_3\text{C}\}(\text{C}_5\text{H}_5)(\text{CH}_3)_2]$ , trigonal  $P31c$ , with  $a = 12.681(3)$ ,  $c = 16.124(5)$  Å,  $V = 2245.5(7)$  Å<sup>3</sup>,  $T = 293$  K,  $Z = 4$ ,  $M_r = 558.47$ ,  $D_x = 1.65$  g cm<sup>-3</sup>,  $F(000) = 1104$ , Mo  $K\alpha$ ,  $\lambda = 0.71073$  Å,  $\mu = 4.91$  mm<sup>-1</sup>,  $R = 0.020$  for 1319 reflections with  $F_o > 4\sigma(F_o)$ ;  $S = 2.18$ . Because of crystal decay, three separate crystals were needed for a full data set. These polar (but achiral) crystals showed apparently differing amounts of inversion twinning, leading to problems in accurately merging the three data sets and refining the structure. These problems are discussed briefly.

### 1. Introduction

The substitution of a mono-anionic cyclopentadienyl ligand (Cp) by an isoelectronic dianionic ligand, still a  $6\pi$ -electron donor, to give a new complex with a reduced charge is a theme of current interest in homogeneous Ziegler–Natta catalysis. To this end, several workers have prepared early transition-metal bent metallocenes containing dianionic ligands (Crowther, Baenziger & Jordan, 1991; Bazan, Schaefer & Bercaw, 1993; Uhrhammer, Crowther, Olson, Swenson & Jordan, 1992); these have been characterized structurally and have proven to be useful in olefin polymerization. We have synthesized Zr-based bent metallocenes containing tribenzylidenemethandiide  $[\text{C}(\text{CHC}_6\text{H}_5)_3]^{2-}$ ,  $\text{TBM}^{2-}$ ) as a  $6\pi$ -electron ancillary ligand (Bazan, Rodriguez & Cleary, 1994) and we have now prepared a tantalum compound to determine the role of this dianionic Cp substitute in reactions of group 5 metallocene-like mol-

ecules. We report here the crystal and molecular structure of the title compound.



### 2. Experimental

The compound was prepared from the trifluoromethanesulfonate salt (TBM)  $\text{TaMe}_2(\text{OSO}_2\text{CF}_3)$ , by reaction with  $\text{CpLi}$  in tetrahydrofuran. The compound is not highly soluble in any usual solvent; X-ray crystals were obtained directly from the reaction by layering the two reagents in a 5 ml narrow vial at 243 K and allowing the reaction to proceed for 5 d. Small red hexagonal bipyramids were formed and a number of these were mounted on glass fibres and coated with epoxy cement to retard air oxidation. Earlier work by Rodriguez and Bazan showed that the crystal system is either trigonal or hexagonal; the most probable space group (which was later confirmed) was  $P31c$  with four molecules in the cell. In such a structure two independent molecules must each lie on threefold axes with disorder involving, at least, the cyclopentadienyl and the two methyl groups. Resolving this disorder in the presence of heavily scattering Ta atoms would clearly require high-quality data and careful analysis.

We began by taking oscillation and Weissenberg photographs (zero- and first-layer lines about  $c$ ). The latter showed trigonal symmetry, Laue group  $\bar{3}1m$ , with small deviations from hexagonal,  $6/mmm$  symmetry; there was no indication of twinning, streaking or diffuse reflections. Intensity data were collected on an Enraf–Nonius CAD-4 diffractometer (Mo  $K\alpha$ ,  $\lambda = 0.71073$  Å) at room temperature ( $\sim 296$  K). An entire sphere, to  $\theta =$

\* Contribution no. 9069.

Table 1. *Experimental details*

Crystal data	
Chemical formula	C <sub>29</sub> H <sub>29</sub> Ta
Chemical formula weight	558.47
Cell setting	Trigonal
Space group	<i>P</i> 31 <i>c</i>
<i>a</i> (Å)	12.681 (3)
<i>V</i> (Å <sup>3</sup> )	2245.5 (7)
<i>Z</i>	4
<i>D<sub>c</sub></i> (Mg m <sup>-3</sup> )	1.65
Radiation type	Mo <i>K</i> α
Wavelength (Å)	0.71073
No. of reflections for cell parameters	25
$\theta$ range (°)	0–10
$\mu$ (mm <sup>-1</sup> )	4.91
Temperature (K)	296
Crystal form	Hexagonal bipyramids
Crystal size (mm)	0.3 max.
Crystal color	Lemon–yellow
Data collection	
Diffractometer	Enraf–Nonius CAD-4
Data collection method	$\omega$ scans
Absorption correction	$\psi$ scans
<i>T<sub>min</sub></i>	0.82
<i>T<sub>max</sub></i>	1.16
No. of measured reflections	8437
No. of independent reflections	1522
No. of observed reflections	1319
Criterion for observed reflections	$F_o > 4\sigma(F_o)$
GOF(merge)	1.29
$\theta_{\max}$ (°)	20
Range of <i>h</i> , <i>k</i> , <i>l</i>	–12 → <i>h</i> → 12 –12 → <i>k</i> → 12 –15 → <i>l</i> → 15
No. of standard reflections	3
Frequency of standard reflections	Every 150 min
Intensity decay (%)	6.2, 5.8 and 8.1
Refinement	
Refinement on	<i>F</i> <sup>2</sup>
<i>R</i>	0.020
<i>wR</i>	0.049
<i>S</i>	2.18
No. of reflections used in refinement	1405
No. of parameters used	173
H-atom treatment	Riding
Weighting scheme	$w = 1/\sigma^2(F_o^2)$
( $\Delta/\sigma$ ) <sub>max</sub>	<0.01
$\Delta\rho_{\max}$ (e Å <sup>-3</sup> )	0.25
$\Delta\rho_{\min}$ (e Å <sup>-3</sup> )	–0.31
Source of atomic scattering factors	SHELXL93 (Sheldrick, 1993)

20°, was collected (–12 ≤ *h* ≤ 12, –12 ≤ *k* ≤ 12, –15 ≤ *l* ≤ 15). Since the crystals decayed somewhat in the beam, we needed three crystals to cover the 8437 reflections within this sphere; all three crystals were well formed hexagonal bipyramids, approximately 0.3 mm in the maximum dimension. Each crystal was used until one of the three check reflections showed a drop in intensity of 20%. The decay was approximately linear to that point and data could perhaps have been collected for longer times on each crystal, at the risk of a larger and less accurate decay correction. The crystals had been grown in Rochester perhaps a month before we began data collection in Pasadena; there was no solvent to be lost from them, but a slow air oxidation may have taken place after we removed them from their argon storage

Table 2. *Fractional atomic coordinates and equivalent isotropic displacement parameters (Å<sup>2</sup>)*

$U_{eq} = (1/3)\sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i \cdot \mathbf{a}_j$				
	<i>x</i>	<i>y</i>	<i>z</i>	<i>U<sub>eq</sub></i>
Ta1	0.0	0.0	–0.0134 (1)	0.040 (1)
C1	0.0	0.0	0.1289 (8)	0.024 (3)
C2	0.0979 (8)	0.1198 (8)	0.1097 (5)	0.038 (2)
C3	0.1032 (9)	0.2376 (8)	0.1314 (6)	0.042 (3)
C4	0.1936 (9)	0.3429 (9)	0.0935 (6)	0.051 (3)
C5	0.2005 (11)	0.4547 (11)	0.1147 (9)	0.080 (4)
C6	0.1272 (14)	0.4603 (13)	0.1712 (8)	0.068 (4)
C7	0.0432 (18)	0.3599 (16)	0.2087 (7)	0.061 (3)
C8	0.0297 (10)	0.2474 (10)	0.1903 (7)	0.051 (3)
C9	–0.0230 (23)	0.1578 (25)	–0.0507 (21)	0.130 (16)*
C11	–0.1274 (17)	0.0604 (18)	–0.0787 (13)	0.034 (7)*
C12	–0.0290 (16)	0.1656 (19)	–0.0416 (14)	0.030 (12)*
C13	0.0805 (13)	0.1902 (19)	–0.0826 (13)	0.029 (7)*
C14	0.0497 (21)	0.1002 (21)	–0.1450 (12)	0.075 (11)*
C15	–0.0787 (21)	0.0200 (20)	–0.1426 (12)	0.060 (9)*
Ta2	2/3	1/3	0.0317 (1)	0.042 (1)
C21	2/3	1/3	–0.1086 (9)	0.052 (5)
C22	0.7885 (8)	0.3588 (8)	–0.0914 (5)	0.040 (2)
C23	0.9014 (8)	0.4699 (8)	–0.1138 (6)	0.039 (2)
C24	0.9092 (9)	0.5507 (10)	–0.1719 (8)	0.047 (3)
C25	1.0202 (14)	0.6526 (11)	–0.1964 (9)	0.068 (4)
C26	1.1277 (2)	0.6749 (13)	–0.1562 (9)	0.071 (4)
C27	1.1205 (9)	0.5905 (11)	–0.0977 (7)	0.059 (3)
C28	1.0098 (9)	0.4927 (9)	–0.0789 (6)	0.054 (3)
C29	0.4752 (13)	0.3027 (14)	0.0587 (9)	0.050 (4)*
C31	0.4697 (24)	0.2404 (25)	0.0776 (16)	0.066 (10)*
C32	0.5234 (29)	0.1952 (27)	0.1304 (20)	0.161 (21)*
C33	0.6080 (30)	0.2907 (29)	0.1781 (16)	0.139 (20)*
C34	0.6066 (25)	0.3950 (24)	0.1547 (15)	0.077 (11)*
C35	0.5211 (22)	0.3639 (23)	0.0926 (14)	0.057 (9)*

\* Isotropic.

atmosphere. The crystals were lightly coated with epoxy cement to retard such oxidation, but 6 months after data collection all had lost their original red color and were lemon yellow. Cell dimensions were measured for all three crystals, based on 25 reflections with  $\theta \approx 10^\circ$ . Including overlaps, check reflections (every 150 min) and 666 intensities collected in order to correct for absorption, we measured 10 802 separate intensities in total. The details are summarized in Table 1.

The merging of the three data sets presented some problems. First, all three sets were corrected separately for decay (number 1, 6.2% in *F*; number 2, 5.8%; number 3, 8.1%) and for absorption, by the  $\psi$ -scan technique. Next, the reflections in set number 2 were re-indexed so as to correspond to the same orientation of the unit cell as in numbers 1 and 3 – that is, so that the relatively small differences in intensity that distinguish  $\bar{3}1m$  from  $6/mmm$  would show the same pattern for all three sets. (For all three sets, merging in  $6/mmm$  was noticeably worse, with GOF's > 5.0, than in  $\bar{3}1m$ , where GOF's are  $\sim 1.3$ .) Then a set of 28 reflections with medium–strong intensities and a wide range of  $\theta$  values, common to sets 1 and 3 and with 20 members in set 2, was chosen and average scale factors were calculated [number 1, 1.00; number 2, 0.894 (20); number 3, 1.006 (20)]. Finally came the problem of anomalous dispersion in the presumed space

Table 3. *Selected geometric parameters* (Å, °)

Ta1—C1	2.294 (14)	C1—C2	1.435 (9)
Ta1—C2	2.429 (8)	C2—C3	1.503 (13)
Ta1—C9	2.24 (3)	C3—C4	1.393 (13)
Ta1—C11	2.35 (2)	C3—C8	1.379 (15)
Ta1—C12	2.35 (4)	C4—C5	1.417 (15)
Ta1—C13	2.38 (4)	C5—C6	1.329 (17)
Ta1—C14	2.39 (3)	C6—C7	1.328 (21)
Ta1—C15	2.38 (4)	C7—C8	1.380 (17)
Ta2—C21	2.262 (15)	C21—C22	1.439 (9)
Ta2—C22	2.435 (9)	C22—C23	1.465 (12)
Ta2—C29	2.300 (13)	C23—C24	1.355 (14)
Ta2—C31	2.29 (3)	C23—C28	1.376 (13)
Ta2—C32	2.39 (7)	C24—C25	1.409 (15)
Ta2—C33	2.45 (4)	C25—C26	1.405 (20)
Ta2—C34	2.39 (4)	C26—C27	1.395 (16)
Ta2—C35	2.29 (5)	C27—C28	1.363 (13)
C1—Ta1—C9	105.5 (8)	C8—C7—C6	121.3 (13)
Ta1—C1—C2	77.5 (6)	C7—C8—C3	120.0 (13)
C21—Ta2—C29	100.9 (4)	C23—C22—C21	126.3 (7)
Ta2—C21—C22	78.9 (7)	C24—C23—C22	124.4 (8)
C3—C2—C1	125.9 (8)	C28—C23—C22	119.6 (8)
C4—C3—C2	117.2 (9)	C28—C23—C24	115.8 (9)
C8—C3—C2	123.7 (10)	C25—C24—C23	123.3 (12)
C8—C3—C4	119.0 (9)	C26—C25—C24	118.6 (13)
C5—C4—C3	117.5 (10)	C27—C26—C25	118.2 (12)
C6—C5—C4	121.6 (11)	C28—C27—C26	119.4 (10)
C7—C6—C5	120.5 (12)	C27—C28—C23	124.5 (10)

group *P*31c, and of assuring that all three data sets were of the same polarity before averaging. Accordingly, we first merged the sets two at a time, reversing the polarity of one for a second trial, and chose the pairings with the smallest goodness-of-fit values. The results, while only marginally significant (the GOF values typically differed by ~2%), were consistent, suggesting that the polarity of set number 2 needed to be reversed relative to numbers 1 and 3. The three data sets were then merged, yielding 1522 independent reflections (including space-group absences); the GOF for this merging was 1.29. Deleting space-group absences gave 1405 reflections, of which 1375 showed  $F_o^2 > 0$  and 1319 showed  $F_o > 4\sigma(F_o)$ . During these averaging processes, variances  $\sigma^2 F_o^2$  were derived from counting statistics plus an additional term  $(0.014F_o^2)^2$ ; variances of the resulting averages  $\bar{F}_o^2$  were based on propagation of error plus another additional term  $(0.014\bar{F}_o^2)^2$ .

A Patterson map confirmed the Ta positions we had found with the earlier data ( $R = 0.16$ ) and reasonable tribenzylidenemethandiide ligands were found in a subsequent difference map (reducing the symmetry from that of *P*6<sub>3</sub>*mc* to *P*31c). After four cycles of refinement, the methyl C atoms appeared as relatively weak peaks, consistent with the 2/3 occupancy we had anticipated; they were surrounded by even weaker peaks which we used to position idealized cyclopentadienyl ligands with 1/3 occupancy. Further refinement was encouraging, but the 'Flack parameter' (Flack, 1983) was *ca* 0.55 – a value that suggested almost complete inversion twinning, which was somewhat at odds with our experiences with the data merging. Accordingly, we returned to the three unmerged sets of data, refin-

ing with each separately; the resulting Flack parameters were 0.45 (3), 0.43 (3) and 0.77 (3). It appeared, then, as though there were different amounts of inversion twinning in the three crystals: almost complete twinning for the first two, but moderate purity for the third. It also appeared that our merging had been incorrect, the third data set having been given the wrong polarity relative to the first two (presuming that the first two numbers are indeed different from 0.5).

At this time we faced a quandary. We could report, as final results, the averages of the three independent refinements, each based on a partial data set; however, the averaging would be questionable in view of the many important covariance terms in this hypersymmetric and disordered structure – terms that could be accurately obtained only by extensive programming efforts. We chose, instead, to presume that after inverting the polarity of the third data set relative to the other two, the three would be sufficiently comparable that they could be combined in a single refinement. Accordingly, we remerged with the revised polarities and completed the refinement, introducing H atoms at calculated positions riding on their attached C atoms (C—H, 0.95 Å;  $U_H = 1.2U_C$ ; the orientations of the methyl groups about the Ta—C bonds were included as refinable parameters). The Ta and C atoms of the tribenzylidenemethandiide ligand were given anisotropic  $U_{ij}$ 's; those of the disordered methyl and cyclopentadienyl groups were held isotropic. The final  $R$  was 0.020 for the 1319 data with  $F_o > 4\sigma(F_o)$  and the GOF was 2.18 for all 1405 independent reflections and 173 parameters (including seven parameters – the orientation, the position of the centroid and the mean C—C distance – for each of the two independent cyclopentadienyl groups). The largest shift/e.s.d. in the final cycle was  $< 0.01$  and maximum excursions in a final difference map were  $+0.25$  and  $-0.31$  e Å<sup>-3</sup>. The final value of the Flack parameter was 0.37 (2) – exactly the average of the three separate values we obtained earlier (taking into account the reversed polarity of data set number 3).<sup>\*</sup> Final heavy-atom parameters are given in Table 2 and final heavy-atom distances and angles are in Table 3.<sup>†</sup> Calculations for data collection and cell refinement were performed with the CAD-4 programs (Enraf–Nonius, 1989) and for preliminary data processing, with the *CRYM* system (Duchamp, 1964). Final data processing and structure solution were performed with *SHELXS86* (Sheldrick, 1990) and refinement with *SHELXL93* (Sheldrick, 1993). Drawings were performed using *ORTEPII* (Johnson, 1976).

<sup>\*</sup> We note that this is true 'inversion twinning' – inversion through a center of symmetry – rather than twinning by reflection across (001), which would have far more important consequences.

<sup>†</sup> Lists of structure factors, anisotropic displacement parameters and H-atom coordinates have been deposited with the IUCr (Reference: BK0026). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

### 3. Discussion

A view of the Ta2 molecule looking down the threefold axis is shown in Fig. 1. Drawings of the two independent molecules in the asymmetric unit are in Fig. 2. Although both lie on threefold axes and hence have the same substitutional disorder, involving the cyclopentadienyl ring and the two methyl groups, their environments are quite different (Fig. 3). The Ta1 molecules lie one above the other along the *c* axis, separated by *c*/2 or 8.06 Å. On the other hand, the Ta2 molecules form zigzag chains, lying alternately at  $(\frac{1}{3}, \frac{2}{3}, z)$  and  $(\frac{2}{3}, \frac{1}{3}, z + \frac{1}{2})$ , the distance between them being 10.89 Å. (The shortest distance between Ta atoms is Ta1...Ta2 at 7.36 Å. A drawing of the two molecules within such a layer, at *z* ≈ 0, is shown in Fig. 4.) The difference in packing of the two molecules seems to have had little influence on the bond lengths and angles or the conformations, although we note that the cyclopentadienyl ring on Ta1 appears to be pinched in (towards the tribenzylidenemethandiide group) more than that on Ta2 (Cp1—Ta1—C1 132, Cp2—Ta2—C21 138°), perhaps to relieve crowding with a neighboring molecule along *c*; concomitantly, the angles to the methyl groups on Ta1 are expanded: 105.5 (8) *versus* 100.9 (4)° at Ta2. However, there are surely large correlations between corresponding distances and angles in the two different molecules, because of the hypersymmetry caused by the Ta atoms lying on special positions, and we have more confidence in average bond lengths and angles than in the individual values given in Table 3. (The largest correlation coefficient, 0.82, is between the isotropic displacement parameters of two disordered atoms — the methyl group C9 and a cyclo-

pentadienyl atom C12, which are separated by only 0.21 Å.) We particularly note that the tribenzylidenemethandiide ligand is appreciably 'domed' (*i.e.* curved toward the Ta atom) in both molecules, with the three methylene C2 atoms (and C22) bent in towards the Ta atoms by ~ 0.3 Å (note that the Ta1—C1—C2 and Ta2—C21—C22 angles are acute). The C2—C1—C2' angle is 115.4 (5)° and C22—C21—C22' is 116.4 (4)°. Thus, the tantalum-tribenzylidenemethandiide bonding can indeed be described as  $\eta^4$ . Distances in the benzyl rings range from 1.328 (21) to 1.417 (15) Å, a somewhat larger spread than expected; the average C—C distance is 1.378 (29) Å, approximately as expected. The benzyl groupings are planar within 0.02 Å; in both molecules they make angles of ~44° with the *c* axis. Average Ta—C(Cp) distances are: for (1) 2.370 (17) Å (range 2.350–2.391 Å) and for (2) 2.363 (72) Å (range 2.288–2.452 Å). The refined C—C distances in the Cp rings are: for (1) 1.42 (2) and for (2) 1.38 (2) Å. We emphasize

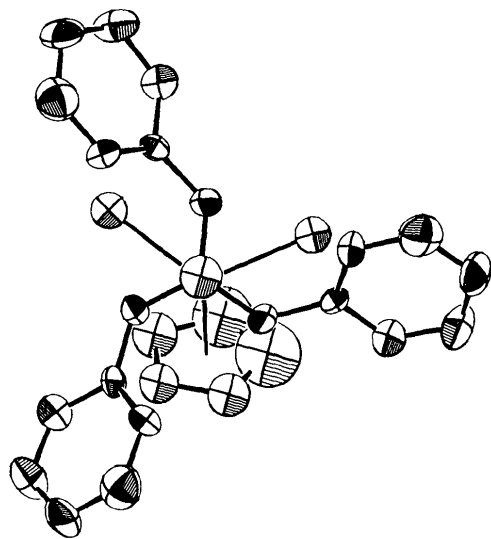


Fig. 1. A view of molecule (2) down the Ta2—C21 bond drawn using ORTEP (Johnson, 1976), with atoms shown as 50% probability ellipsoids. H atoms are not shown. Only one position of the disordered Cp ring and methyl groups is shown.

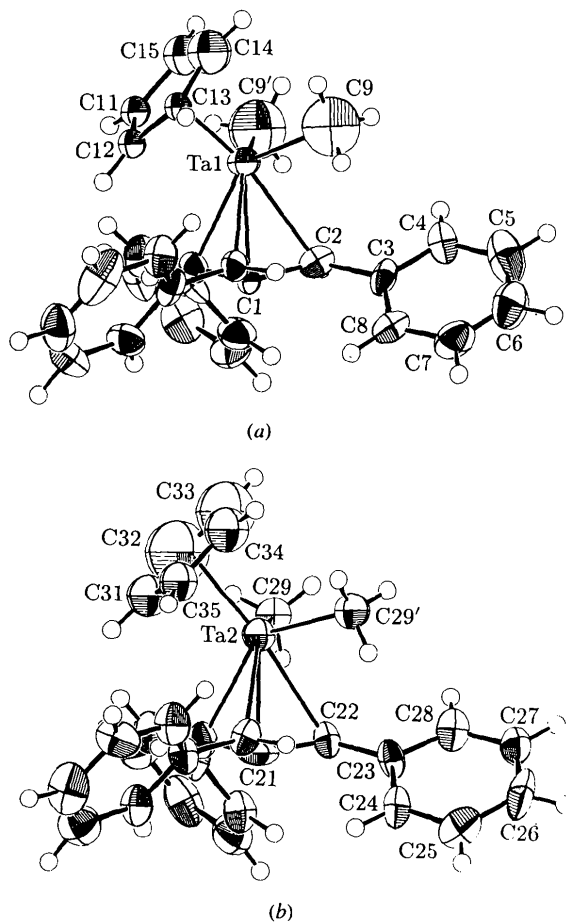


Fig. 2. ORTEP (Johnson, 1976) drawings of the two independent molecules with 50% probability ellipsoids, showing the numbering system. H atoms are shown as spheres of arbitrary, small radii. Only one position of the Cp ring is shown; the ring and the two methyl groups are disordered about the threefold axis passing through Ta1 and C1 or Ta2 and C21.

that the parameters of the Cp rings have not been well determined, not only because of their low population factors (the sites are only one-third occupied, due to the disorder), but also because of overlap with the methyl groups C9 and C29. The  $U$  values of these atoms (Table 2) are particularly suspect; we might have chosen to constrain them in some way, but did not.

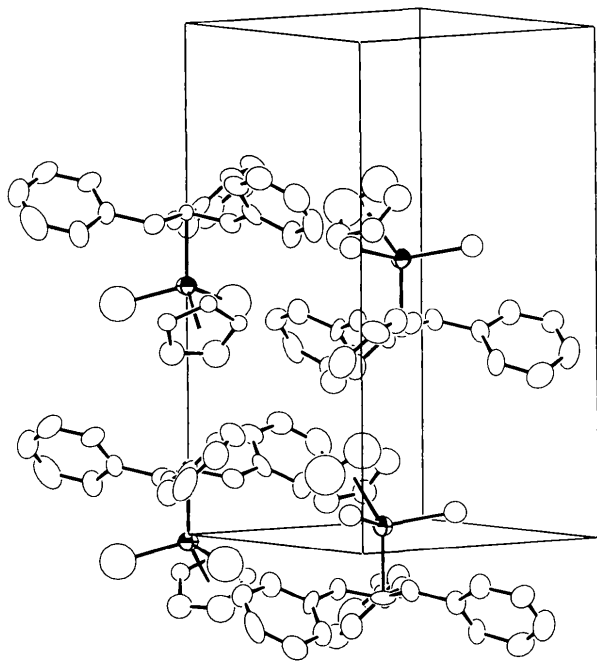


Fig. 3. An ORTEP (Johnson, 1976) drawing of the contents of a unit cell, with a unit cell outlined. Atoms are shown as 50% probability ellipsoids; H atoms are not shown. The view is approximately parallel to the  $ab$  plane; the  $c$  axis is vertical. The Ta atoms are shown with shaded octants.

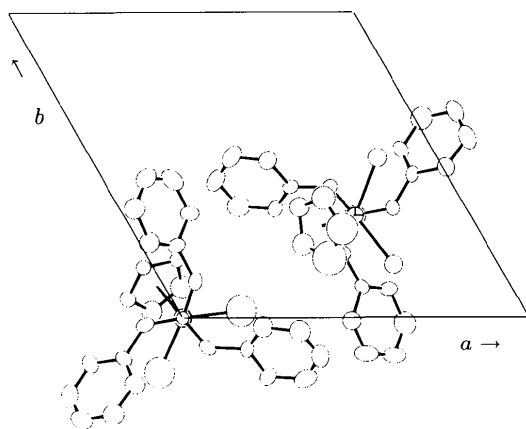


Fig. 4. An ORTEP (Johnson, 1976) drawing projected down the  $c$  axis showing the outline of the unit cell and an asymmetric unit of atoms. The Ta atoms shown have  $z$  coordinates of approximately 0; a second 'layer' at  $z \sim \frac{1}{2}$  has the second Ta1 molecule directly above the first at  $0, 0, \frac{1}{2} + z$  and the second Ta2 molecule displaced from the first at  $\frac{1}{3}, \frac{2}{3}, \frac{1}{2} + z$ .

In view of the rather peculiar packing that we find in this structure, with the two independent molecules required (by symmetry) to have quite different environments, we have searched the Cambridge Structural Database (Allen, Kennard & Taylor, 1983; October 1994 release) for other examples of space group  $P31c$ . There are only 26 such entries (out of a total of 126 353), of which only one, iron tris(*N*-methyl, *N*-butyl-dithiocarbamate) (Terzis, Filippakis, Mentzafos, Petrouleas & Malliaris, 1984), has  $Z = 4$ , with the metal atoms in the same set of special positions as in this tantalum complex. This iron compound has threefold molecular symmetry and the structure shows no disorder; however, the *n*-butyl groups have different conformations in the two molecules. [We further note that throughout the original paper (Terzis, Filippakis, Mentzafos, Petrouleas & Malliaris, 1984), the space group is referred to as  $P3_1/c$  rather than  $P31c$ .]

Finally, we note that although the space group  $P31c$  requires that the structure be polar, the two molecules in the asymmetric unit point in opposite directions (along  $c$ ). Accordingly, if polar dispersion errors (Cruickshank & McDonald, 1967) remain as a result of our merging data sets with different amounts of inversion twinning, these errors should be in the opposite sense in the two molecules and once again the average bond lengths should be reliable. However, we remain somewhat uncomfortable with the procedure we used, which would have been even more inappropriate if one of the three data sets had been appreciably more extensive, or appreciably more accurate, than the others. We suggest that authors of computing packages consider including options to handle correctly the case where two or more crystals having differing amounts of inversion twinning are to be incorporated into a single structure analysis.

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